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Environmental and Chemical Factors Associated with Maple Sugar Sand Formation

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Environmental and Chemical Factors Associated with Maple Sugar Sand Formation[1]

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INTRODUCTION

The maple syrup industry is truly American, for the first settlers of this nation learned the art of making maple syrup from the Indians. With its distinctive flavor and sweet taste, maple syrup has long been a popular food item for the American people.

The major maple syrup production areas of the United States are concentrated near the Great Lakes region and the St. Lawrence River. Ohio, with its vast stands of maple trees, is a major producer of maple syrup and is second only to New York and Vermont. The principal syrup producing area is the northeastern section of the state. In this region, Geauga County leads all Ohio counties with a production of approximately one-third of the state's total.

Although this industry is one of the oldest in the United States, relatively little scientific research has been conducted to improve its production until recently. This investigation is directed to one of the most important and costly problems of the maple syrup producer—maple sugar sand. This gritty material, also called niter and lime, is formed in maple syrup during the concentration (boiling) of sap to syrup.

Sugar sand appears as a precipitate which is usually suspended in maple syrup. While the syrup is still in the evaporator, this precipitate is partially deposited as a scale on the walls of the evaporator pan. This causes a reduction in the evaporator efficiency since it decreases the transmission of heat to the boiling syrup. The layer of sugar sand scale also tends to lower the grade (color) of the syrup by making it darker. This results from the caramelized sugar being trapped in the scale layer on the evaporator.

The maple sugar sand remaining in suspension causes the syrup to be cloudy. To meet Federal and most state standards of table maple syrup, all syrup must be clarified to provide a clear product (free from cloudiness or suspended matter). Suspended sugar sand can be removed from the maple syrup by filtration or sedimentation. The removal of sugar sand by either of these methods is both expensive and laborious.

Earlier investigators of maple sugar sand directed their research towards its chemical composition (2, 5, 6, 8). Today, sugar sand has been recognized as being principally a calcium malate precipitate (7, 9). Willits (9) states that the calcium salts of malic acid are precipitated because they become less soluble as the temperature of the syrup solution increases and as its concentration increases.

Among the first investigators of sugar sand composition were Huston and Bryan (5), who found that calcium and malic acid were the major constituents. Later, Warren (8) collected sugar sand samples from different localities and reported them to be composed of 17.2 percent calcium and 51.5 percent malic acid.

A detailed study of the acids in maple sugar sand was made by Nelson (6). He separated and identified the following; 1-malic, formic, acetic, fumaric, succinic, and citric. Davis et al (2) showed recently that the amount of sugar sand precipitated was directly related to the relative amounts of calcium malate in sugar sand.

Although calcium and malic acid have been shown to be the major constituents and cause of sugar sand deposition, little attention has been given to their variability and degree of influence on sugar sand formation. It might be expected that certain ecological conditions may be associated with their effect on the amount of sugar sand formed. Therefore, this investigation was conducted to determine the effect of some chemical and ecological factors upon the formation of maple sugar sand.

The ecological factors studied were: exposure of the sugar bush site, soil texture, sugar bush elevation, air temperature, and precipitation. The study included both chemical and microscopic analyses of sugar sand samples obtained from 32 syrup producers in Ohio. Each chemical constituent was related to the amount of sugar sand formed during the concentration of sap to syrup.

Maple Syrup Cooperators

Thirty-two maple syrup producers, located throughout the maple syrup area in Ohio, cooperated in this study (Fig. 1). These 32 collaborators were selected because their sugar bushes included all ecological factors suspected of influencing sugar sand formation. These ecological factors included four different exposures (north, south, east, and west); two soil types (heavy and light); and two elevations (high and low). These conditions were met by a factorial designed experiment, 4 x 2 x 2, which provided all data necessary for statistical analysis.

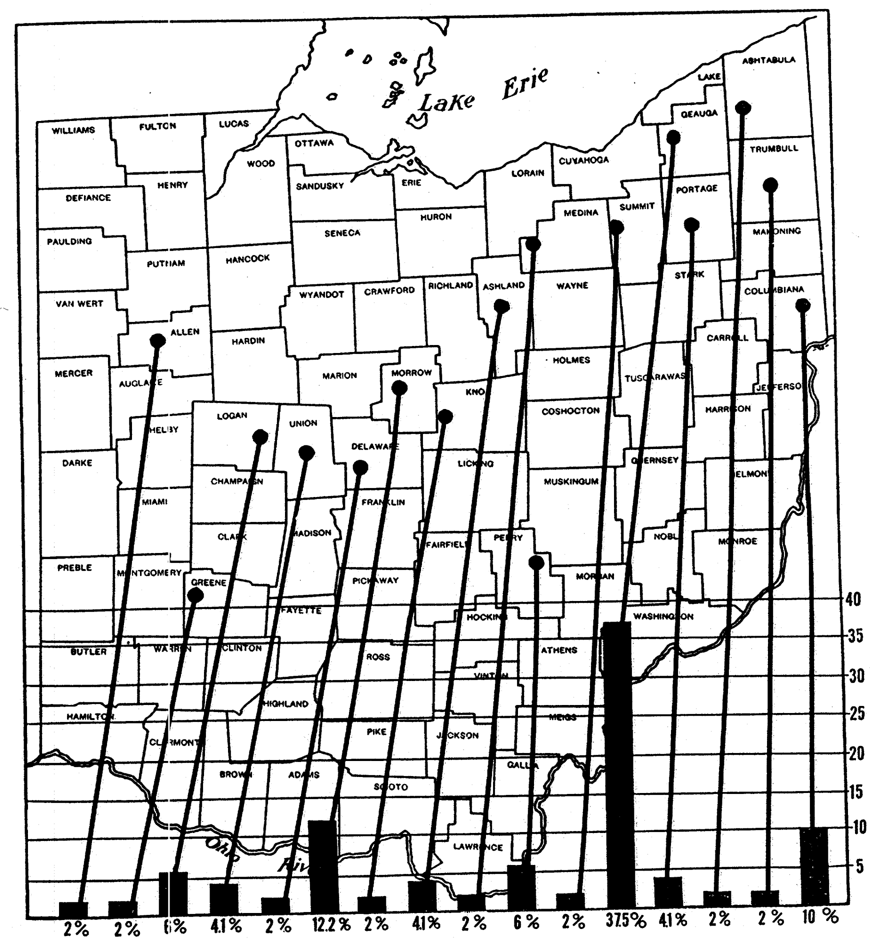

Fig. 1.—Distribution of maple syrup cooperators in Ohio by counties.

During each of the 4 years of this investigation (1960 through 1963), the cooperators were contacted prior to the beginning of the maple syrup season. They were given specific instructions and supplies necessary for recording data and collecting maple syrup and sugar sand samples.

Maple Sugar Sand Samples

Three maple sugar sand samples were obtained each year from each collaborator and represented syrups made during the early, middle, and late sap flow periods of each season. The samples represented the filtrable material taken from 5 or more gallons of syrup made during the process run (sap flow period).

Each sugar sand sample was removed from the filter and placed in a polyethylene bag. At the time the sugar sand sample was taken, the volume of syrup from which it had been obtained was noted and a sample of the syrup was taken. Both the sugar sand and the syrup samples were taken by the producers. These were collected at weekly intervals and delivered to the Department of Horticulture laboratories at the Ohio Agricultural Research and Development Center, Wooster. The samples were held under refrigeration (32° F.) until analyzed.

The total number of process runs with the corresponding sugar sand and syrup samples from the 32 producers are shown in Table 1. The column headed "Number of Process Runs with Maple Sugar Sand" lists those samples which were chemically analyzed each year of this study.

In a few instances the process runs of syrup contained insufficient amounts of material to yield a measurable amount of sugar sand when filtered (column 2). Therefore, no sugar sand samples were obtained or recorded for these process runs. Such runs, however, were classified as an observation and were included in the total number of process runs. In addition, these runs were used in the calculation of data and were represented as 0 percent sugar sand.

Analytical Methods

Following the 32° F. storage period, each crude sugar sand sample was removed from the polyethylene bag and uniformly mixed by stirring for 10 minutes. Two subsamples were then taken, one for the determination of sugar and the other for the determination of total solids. The dried samples obtained from the total solids determination were used for the inorganic, malic acid, and total organic acid analyses.

The total solids of the samples were determined by the Vacuum Drying Method for maple syrup of the A.O.A.C. Methods of Analysis (1). The total sugars were determined after acid hydrolysis by the

TABLE 1.—Total Number of Samples and Process Runs with and without Maple Sugar Sand of Various Maple Syrup Producers, 1960 to 1963.

Year	Number of Process Runs with Maple Sugar Sand	Number of Process Runs without Maple Sugar Sand	Total Number of Process Runs
1960	32	3	35
1961	83	4	87
1962	85	11	96
1963	89	7	96

A.O.A.C. Method, the Quantitative Determination of Reducing Sugars (1). Sugar content was calculated as percent total reducing sugars.

The methods used to determine malic acid and total organic acids were reported previously by Davis et al (2).

The total calcium, magnesium, and potassium content was determined by flame analysis, using a Beckman D U spectrophotometer with a flame attachment. The analysis was performed on the extracted solution of the dried sugar sand samples. The flame emission spectra were measured at the following wave lengths: 422 millimicrons (calcium), 285 millimicrons (magnesium), and 767 millimicrons (potassium).

The degrees Brix of each lot of processed syrup from the various process runs were determined by measuring the refractive index of the syrup, using an Abbe refractometer.

Percent Maple Sugar Sand

The maple sugar sand samples received from the maple syrup producers were designated "as received" or crude samples. For the purposes of this investigation, the amount of maple sugar sand in the crude samples was determined by subtracting the weight of the sugar from the weight of total solids of each sample. Then the percent maple sugar sand in each process run was calculated by first determining the weight of maple sugar sand in the crude sample and then dividing into this weight the total weight of the process run (weight of syrup + weight of the crude sample) and multiplying by 100. This is represented by the following equation:

$$\text{Percent Maple Sugar Sand in Syrup of Each Process Run} = \frac{X}{Y+Z} \times 100$$

X = weight of maple sugar sand in the crude sample

Y = weight of syrup from which the crude maple sugar sand was obtained

Z = weight of the crude sample

EXPERIMENTAL RESULTS

Percent Maple Sugar Sand

The percentages of maple sugar sand in the syrup from each of the different process runs are summarized in Table 2. The percent of maple sugar sand formed in the different syrups produced in the same year varied widely, as shown by the high standard deviations (Table 2). The greatest range occurred in 1961, with a variation between 0.00 and 2.63 percent. Although the variability was high, a trend was obtained for the sand produced during the early, middle, and late process runs. For each year of this study, the percent of maple sugar sand increased as the syrup season progressed.

Comparing yearly averages of percent of maple sugar sand in syrup, a definite reduction in the percentage was observed in 1962. The average percentage of sugar sand in syrup was 0.13 percent, which was less than half that of the other 3 years.

Chemical Analyses

The results of the chemical analyses of the maple sugar sand samples are summarized in Tables 3 and 4. The range and mean values with a standard deviation of each constituent were determined from the data obtained for each year of this investigation. The summary data for total sugars and total solids of the "as received" or crude samples are shown in Table 3.

The average percentage of total sugars and total solids showed only small variations for the 4 years, varying between 50 to 54 percent and 66 to 70 percent, respectively. However, the range of values of the two constituents showed that the data for the different process runs varied greatly within each year. The percent of total sugars for 1960 had the greatest variability, with minimum and maximum values of 4.40 and 85.22 percent, respectively. The variation of the percent total solids was also greatest in 1960, with a range of 25.15 to 86.51 percent. Although an extreme range existed for both constituents, the summarized results of the data show that the majority of the values are grouped around the yearly averages, accounting for the relatively low standard deviation values.

Chemical analyses of maple sugar sand revealed that malic acid was the major constituent. The average malic acid content (Table 4) for each of the 4 years varied between 6.78 and 11.30 percent. The minimum and maximum and the standard deviation of the percent malic acid indicate that this constituent varied the greatest in 1961, ranging between 0.49 and 46.49 percent.

TABLE 2.—Percent Maple Sugar Sand in Syrups of Different Process Runs, 1960 to 1963.

Process Run	Average Percent Maple Sugar Sand in Syrups by Years			
	1960	1961	1962	1963
Early	0.20	0.22	0.09	0.22
Middle	0.34	0.35	0.14	0.28
Late	0.45	0.46	0.16	0.30
Minimum	0.00	0.00	0.00	0.00
Maximum	1.30	2.63	0.99	2.27
Number of Samples	32	83	85	89
Yearly Average	0.30	0.34	0.13	0.27
Standard Deviation	0.38	0.37	0.28	0.32

TABLE 3.—Summary of Total Sugars and Total Solids Analyses of Maple Sugar Sand Samples Received from Various Cooperators, 1960 to 1963.

Year	Content	Number of Samples	Composition of Maple Sugar Sand by Years	
			Total Sugars Percent	Total Solids Percent
1960	Minimum	32	4.40	25.15
	Maximum		85.22	86.51
	Average		49.86	70.08
	Standard Deviation		18.07	12.13
1961	Minimum	83	4.34	40.61
	Maximum		72.64	81.15
	Average		53.79	68.52
	Standard Deviation		19.64	9.30
1962	Minimum	85	6.90	29.23
	Maximum		76.84	82.79
	Average		53.19	65.84
	Standard Deviation		17.02	11.31
1963	Minimum	89	11.92	28.42
	Maximum		69.32	78.50
	Average		53.43	68.26
	Standard Deviation		14.54	9.50

TABLE 4.—Chemical Analyses on a Dry Weight Basis of Maple Sugar Sand Samples Received from Various Cooperators, 1960 to 1963.

Year	Number of Samples	Content	Percentage of Chemical Constituents in Dried Maple Sugar Sand Samples					
			Ca	K	Mg	Organic Acids	Malic Acid	Acids Other Than Malic
1960	32	Minimum	0.30	0.09	0.00	0.84	0.48	0.02
		Maximum	7.80	7.80	0.17	29.62	25.18	11.71
		Average	3.01	0.29	0.04	10.11	8.70	1.43
		Standard Deviation	2.83	0.06	0.02	9.63	8.31	2.54
1961	83	Minimum	0.45	0.04	0.01	0.50	0.49	0.01
		Maximum	11.58	0.40	0.19	48.52	46.49	2.62
		Average	3.98	0.25	0.03	11.94	11.30	0.64
		Standard Deviation	3.27	0.08	0.02	11.81	11.37	0.62
1962	85	Minimum	0.28	0.05	0.00	0.64	0.54	0.01
		Maximum	11.50	0.29	1.45	38.08	37.01	2.23
		Average	2.61	0.17	0.17	7.25	6.78	0.47
		Standard Deviation	2.96	0.07	0.25	9.32	8.90	0.51
1963	89	Minimum	0.05	0.02	0.01	0.27	0.16	0.02
		Maximum	10.65	1.51	0.17	33.37	32.55	6.86
		Average	2.52	0.21	0.06	8.25	7.15	1.09
		Standard Deviation	2.15	0.19	0.03	6.70	6.26	1.42

The acids other than malic were generally unimportant since malic acid accounted for 85 to 95 percent of the total acids present. The largest average content of acids other than malic, 1.43 percent, was obtained for the year 1960 (Table 4). However, in 1960 and 1963 these acids reached a maximum value of 11.71 and 6.86 percent, respectively.

The average percentage of calcium in maple sugar sand samples for each year of this study was second to that of malic acid (Table 4). The yearly calcium average varied between 2.52 percent and 3.98 percent, with very large differences in percentages for the samples over the 4 years. The largest percentage, 11.58 percent, occurred in 1961 and the smallest percentage, 0.05 percent, in 1963. The high standard deviations for calcium indicate that extreme variations in content of the samples occurred within each year.

The contents of potassium and magnesium in maple sugar sand were considerably less than that of calcium (Table 4). The average potassium and magnesium contents for the 4 years were 0.23 and 0.07 percent, respectively.

Chemical Constituents and Percent of Maple Sugar Sand Formed

The correlation coefficients of the chemical constituents with the percent maple sugar sand formed during each process run are shown in Table 5. A definite positive relationship was found between the percent sugar sand formed and the calcium and malic acid contents of the samples. As the contents of both constituents increased in maple sugar sand, there was an increase in the amount of sugar sand formed.

Since malic acid was found to be the major acid in maple sugar sand (Table 4), a definite positive correlation was also found between total organic acids and percent sugar sand formed (Table 5). The correlation between percent maple sugar sand and percent acids other than malic was significant for only 2 years (1961 and 1962).

Since highly significant positive correlations were obtained between percent maple sugar sand formed and the calcium and malic acid contents, the data were analyzed independently of one another and independently of the yearly variation by least squares analysis of variance. The results, Table 6, indicate that the calcium effects were significant but that the malic acid effects were not.

The relationship of calcium and maple sugar sand present in each process run was determined by regression techniques. The general linear regression equation is as follows: percent maple sugar sand $=$ $.0703 + .0481$ (percent calcium) $+ .0036$ (percent malic acid). Average values for percent maple sugar sand, calcium, malic acid were: .2495, 3.0763 and 8.6691, respectively. This shows that an increase of

TABLE 5.—Correlation Coefficients Relating Chemical Constituents in Maple Sugar Sand and Percent Maple Sugar Sand Formed During Each Process Run, 1960-1963 Seasons.

Constituents Correlated	Correlation Coefficients			
	1960	1961	1962	1963
% Maple Sugar Sand vs. % Calcium	.629**	.594**	.471**	.524**
% Maple Sugar Sand vs. % Potassium	—.049	—.619**	—.059	—.053
% Maple Sugar Sand vs. % Magnesium	—.192	.069	—.399**	—.022
% Maple Sugar Sand vs. % Total Malic Acid	.689**	.508**	.466**	.492**
% Maple Sugar Sand vs. % Total Organic Acids	.611*	.516**	.475**	.486**
% Maple Sugar Sand vs. % Acids, Other Than Malic	.062	.520**	.545**	.180

*Significant at the 5% level.
**Significant at the 1% level.

TABLE 6.—Analysis of Variance of Percent Maple Sugar Sand for Yearly Effects and Regressions on Calcium and Malic Acid.

Source	Sum of Squares	df	MS	F	Significant at
Years	.4634	3	.1545	2.303	.10
Calcium	.7505	1	.7505	11.183	.01
Malic Acid	.0437	1	.0437	1	N.S.
Residue	19.6774	293	.0671		

1 percent calcium is associated with an increase of .05 percent in maple sugar sand.

Microscopic Examination

Microscopic examination of the sugar sand showed the presence of two crystal forms (Figure 2). Those in greatest amount were of rhomboidal form, opaque to clear, with an average size of 2 x 10^{-2} mm. x 2 x 10^{-3} mm. Those in lesser amount were cubical in form with an average size of 5 x 10^{-3} mm. x 5 x 10^{-3} mm. The major portion of sugar sand as viewed under the microscope appeared as amorphous material resulting from abnormal crystal growth.

Fig. 2.—Photomicrograph of maple sugar sand showing crystalline and amorphous materials.

Ecological Factors and Percent of Maple Sugar Sand Formed

The data relating the effect of different ecological factors of sugar bushes to the production of maple sugar sand in syrup were compiled over a 4-year period, 1960-1963. The data in Table 7 relate the amounts of sugar sand formed to: exposure (cardinal compass location) of the sugar bush, sugar bush elevation, and soil texture.

The results indicate no interaction between elevation and eastern exposure and the amount of sugar sand formed. Those sugar bushes of western and southern exposure produced more sugar sand at high elevation. The reverse was observed for sugar bushes with northern exposure, where the most sugar sand was formed at lower elevations. The interaction of soil texture and exposure on amount of sugar sand formed revealed that bushes grown on heavy soils with southern exposure and on light soil with northern exposure produced the greatest amount of sugar sand.

The interaction of the relative soil textures and the elevation of the sugar bushes indicates no real effects for light soils. However, significantly smaller amounts of maple sugar sand were formed in syrup of sugar bushes on heavy soils at lower elevations.

TABLE 7.—Mean Percentages of Maple Sugar Sand Formed in Syrup as Related to Ecological Factors, 1960 to 1963.

Exposure	Elevation: High	Elevation: Low
North	.196	.394
East	.189	.271
South	.484	.117
West	.240	.079
L.S.D. at 0.05% = 0.136		

Exposure	Soil Texture: Heavy	Soil Texture: Light
North	.209	.381
East	.150	.283
South	.351	.250
West	.154	.164
L.S.D. at 0.05% = 0.136		

Soil Texture	Elevation: High	Elevation: Low
Heavy	.641	.221
Light	.540	.539
L.S.D. at 0.05% = .096		

Data concerning the minimum and maximum monthly temperatures and the amount of precipitation for each month and quarter year were statistically correlated with the percent of sugar sand formed. The results showed that the October minimum mean temperature and amount of rainfall during the second quarter (April-June) were significantly associated with percent sugar sand. In addition, regression analysis provided results which indicated that the second quarter rainfall was negatively correlated with percent sugar sand while the October minimum mean temperature was positively correlated. An increase of 1 inch of rainfall during the second quarter reduced the percent sugar sand formed by 0.045 percent. In terms of the October minimum mean temperature, a rise of 1 degree increased the percentage of sugar sand deposited by 0.025 percent.

DISCUSSION

The two major constituents of maple sugar sand are calcium and malic acid (Table 4). These results substantiate the conclusion of past research workers (2, 5, 6, 8). However, most of the previous studies found that calcium and malic acid varied within narrow limits. This did not prove to be true in this study. The variations for the 4 years were 0.05 percent to 11.58 percent and 0.16 percent to 46.49 percent for calcium and malic acid, respectively. These wide variations could have been due to the greater number of maple sugar sand samples included in this investigation.

Although calcium malate has been shown to be the major factor in the formation of maple sugar sand (2, 5, 8), the data showed that the precipitated material of the "as received" samples was comprised largely of sugar. The average sugar content was approximately 53.0 percent of the crude maple sugar sand weight. Even though sugar was the predominant constituent, it was not regarded as being important in the deposition of maple sugar sand, since sugar (sucrose) is readily soluble at the density of maple syrup (65.46° Brix).

Although sugar is not the direct cause of maple sugar sand formation, it is an important factor in the quantity of maple syrup obtained from a given amount of sap. Its entrapment in maple sugar sand causes an average loss of approximately 1.5 percent of the amount of syrup produced in each process run (3). The average loss is usually not great but losses of syrup yields can be as high as 10 percent.

The results of this study confirmed and extended previous findings that calcium and malic acid influence the amount of maple sugar sand formed during sap to syrup concentration. This relationship was found to be positive and significant and suggests that calcium malate is the principal organic salt in maple sugar sand. However, the effect of calcium was greater than that of malic acid (Table 6). This indicates that less calcium than malic acid is needed for the formation of an equal amount of maple sugar sand. This difference may be explained by comparing the percentage of each constituent in calcium malate. The percentage of calcium in calcium malate (molecular weight 172.15) is 23.28 percent, compared with malic acid of 76.72 percent.

Data showed that the amount of sugar sand formed varied from year to year within and between producers (Table 2). So a syrup producer could expect a variation in the amount of sugar sand formed during the processing season and also between seasons. Although the amount varied widely, some producers had less sugar sand than others. This study established that less sugar sand was obtained from sites with a southern or western exposure at relatively low elevations. This suggests that syrup quality could be improved by site selection.

Since the amount of sugar sand varies between producers and seasons and within each season, it is apparent that some factor or factors influence the amount of calcium and malic acid for sugar sand deposition. Further, the amount of sugar sand formed was affected by soil texture, elevation, and exposure. This suggests that these ecological conditions are also associated with calcium and malic acid.

Differences in soil composition (calcium content), soil moisture, and rate of calcium absorption by the tree root system may affect sugar sand formation. Calcium absorption appears to be associated with soil temperature, which varies between sugar bush sites of different elevations and exposures and also during the processing season. A sugar bush on a southern exposure and at relatively high elevation would be exposed to a greater period of direct sunlight and higher temperatures. This could result in greater calcium absorption. Data from this study (Table 2) appear to substantiate this supposition, since more sugar sand was formed as the syrup season progressed and temperatures increased.

Malic acid production may also be associated with temperature and sunlight, since this acid is synthesized through a series of metabolic reactions which are temperature- and light-related. Thus, differences in temperature and sunlight during the syrup season and at various elevations and exposures may be responsible for variations in amount of malic acid produced in sugar sand formation.

Although the content of calcium and malic acid in maple sap may be associated with certain ecological conditions, the possibility that other factors have an effect on sugar sand formation cannot be excluded. Davis et al (2) showed that the content of iron, copper, and boron in sugar sand was negatively correlated with the amount formed during syrup processing. This suggests that these constituents may affect the formation of sugar sand by either limiting the uptake of calcium through the root system or affecting the mechanism of calcium malate precipitation during boiling.

Differences in syrup processing procedures also may vary the amount of sugar sand formed. Since sugar sand is believed to be a calcium malate precipitate, factors such as time and temperature of heating, amount of agitation, and rate of settling can affect the character of the precipitate. Thus, differences in processing may vary the size, shape, and form of the calcium malate precipitate and therefore affect the quantity of sugar sand formed.

SUMMARY

Highly significant positive correlations were obtained between the percent sugar sand formed and the calcium and malic acid content. However, evidence was found that calcium was more critical in sugar sand deposition than malic acid.

The major part of the crude sugar sand consists of amorphous material, together with small amounts of rhomboidal and cubical crystals which are both clear and opaque.

With regard to the ecological factors, there were significant effects of the interactions of exposure and elevation, soil texture and exposure, and soil texture and elevation.

The ecological data suggest that syrups with lower sugar sand content will be obtained from sugar bushes having a southern or western exposure at a relatively low elevation. The data also suggest that the texture of the soil is not important.

Temperature effects may be responsible for variations in the amounts of sugar sand formed as related to the other ecological factors of a given sugar bush.

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The State Is the Campus for Agricultural Research and Development

Ohio's major soil types and climatic conditions are represented at the Research Center's 11 locations. Thus, Center scientists can make field tests under conditions similar to those encountered by Ohio farmers.

Research is conducted by 13 departments on more than 6000 acres at Center headquarters in Wooster, nine branches, and The Ohio State University.

Center Headquarters, Wooster, Wayne County: 1918 acres
Eastern Ohio Resource Development Center, Caldwell, Noble County: 2053 acres
Mahoning County Experiment Farm, Canfield: 275 acres
Muck Crops Branch, Willard, Huron County: 15 acres
North Central Branch, Vickery, Erie County: 335 acres
Northwestern Branch, Hoytville, Wood County: 247 acres
Southeastern Branch, Carpenter, Meigs County: 330 acres
Southern Branch, Ripley, Brown County: 275 acres
Vegetable Crops Branch, Marietta, Washington County: 20 acres
Western Branch, South Charleston, Clark County: 428 acres